

MIL-B-387,1(USAF)
30 July 1965

MILITARY SPECIFICATION

BROMOCHLORODIFLUOROMETHANE, TECHNICAL

1. SCOPE

1.1 This specification covers the requirements for bromochlorodifluoromethane for use as a fire extinguishing agent.

2. APPLICABLE DOCUMENTS

2.1 The following standards of the issue in effect on date of invitation for bids form a part of this specification to the extent specified herein:

STANDARDS

Federal

Federal Test Method Standard
No. 791

Lubricants, Liquid Fuels and
Related Products; Methods of
Testing

Military

MIL-STD-101

Color Code for Pipelines and
for Compressed Gas Cylinders

MIL-STD-105

Sampling Procedures and
Tables for Inspection by
Attributes

MIL-STD-129

Markings for Shipment and
Storage

(Copies of specifications, standards, drawings and publications required by suppliers in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer).

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2.2 Other Publications. The following documents form a part of this specification to the extent specified herein. Unless otherwise indicated, the issue in effect on date of invitation for bids shall apply:

Code of Federal Regulations

Title 49--Transportation
Parts 71-78

Interstate Commerce Commission
Rules and Regulations for the
Transportation of Explosives
and Other Dangerous Articles

(Copies of the regulation cited may be obtained from the Superintendent of Documents, United States Government Printing Office, Washington 25, DC).

American Society for Testing and Materials

ASTM D 1209

Color of Clear Liquids (Platinum-Cobalt Scale)

ASTM 2108

Color of Halogenated Organic
Solvent and Their Admixtures
(Platinum-Cobalt Scale)

(Copies of ASTM publications may be obtained from the American Society for Testing and Materials, 1916 Race Street, Philadelphia 3, Pa.).

3. REQUIREMENTS

3.1 Material. The material shall be a technically pure grade of bromochlorodifluoromethane, suitable as a fire extinguishing fluid and shall conform to the requirements of Table I.

TABLE I - Requirements for Bromochlorodifluoromethane

Property	Requirement	Test Par.
Bromochlorodifluoromethane percent by volume, minimum	99.0	4.4.1
Boiling Point, degrees Centigrade at 760 mm Hg	-4 \pm 1 (24.8 \pm 1.8°F)	4.4.7
Acid Halides & Free Halogens, ppm (by weight), maximum	3.0	4.4.2 and 4.4.3
Nonvolatile Residue, grams/100 ml, maximum	0.02	4.4.4
Suspended Matter or Sediment	None	4.4.5
Color (Platinum-Cobalt Color Standard)	Equal or Less Than #15	4.4.6
Moisture, percent by weight, maximum	0.02	4.4.8

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4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for Inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified, the supplier may utilize his own or any other inspection facilities and services acceptable to the Government. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure supplies and services conform to prescribed requirements.

4.1.1 Inspection shall be in accordance with Method 9601 of Federal Test Method Standard No. 791.

4.2 Sampling for Lot Acceptance.

4.2.1 Inspection Lot. Bromochlorodifluoromethane manufactured as one batch and offered for delivery at one time shall be considered a lot for purposes of inspection.

4.2.2 Sampling for Inspection. A random sample of filled containers shall be selected in accordance with Standard MIL-STD-105 at inspection level I and acceptable quality level (AQL) of 2.5 percent defective to verify compliance with this specification in regards to fill, closure, marking and other requirements not involving tests.

4.2.3 Sampling for Tests. From each lot of filled containers not less than two containers shall be taken at random for tests described under 4.4. The containers may be returned to the lot after acceptance.

4.3 Examination

4.3.1 Filled Containers and Preparation for Delivery Requirements. Each sample selected in accordance with 4.2.2 shall be visually examined for defects of construction of the containers and closure, for evidence of leakage, for unsatisfactory markings and all other preparation for delivery requirements of Section 5. Each sample filled container shall also be weighed to determine the amount of contents. Any container in the sample having one or more defects, or under the required fill, shall be rejected, and if the number of defective containers exceeds the acceptance number for the appropriate sampling plan of Standard MIL-STD-105, the lot represented by the sample shall be rejected. Rejected lots may be resubmitted for acceptance, provided contractor has removed or reworked all nonconforming products.

4.4 Test. Test shall be conducted as specified herein.

4.4.1 Assay. The percentage of bromochlorodifluoromethane shall be determined by gas-liquid chromatography. The components of the sample are separated and the area of each peak measured. The bromochlorodifluoromethane content of the sample is found by comparing the area of its peak with the total area of all the components (See 6.3).

4.4.1.1 Apparatus. The following apparatus is required to determine the percentage of bromochlorodifluoromethane:

- a. Gas chromatograph.
- b. Microsyringe, capable of delivering a 2-microliter sample.

4.4.1.2 Reagents. The carrier gas shall be a commercial grade of helium. The column packing shall consist of 20% (wt/wt) practical hexadecane on 30-60 mesh Chromosorb R or equivalent.

4.4.1.3 Operating Conditions.

a. Column, 12 foot copper tubing 1/4" O.D., packed with reagent.
(See 4.4.1.2).

- b. Column temperature, 40°C
- c. Helium pressure, 3 psi
- d. Helium flow rate, 20 cc/minute
- e. Detector voltage, 8 volts
- f. Recorder span, five m.v. full scale

4.4.1.4 Procedure. Prepare the chromatograph for the conditions specified in 4.4.1.3 and allow it to reach equilibrium so that the base line remains on zero at recorder range setting of one. Cool at 10 ml Erlenmeyer flask in a one pint Dewar flask, filled with dry ice, and transfer two ml of the sample into it. In a second Dewar flask, place the syringe and cover it with dry ice. Allow syringe to cool for 10 minutes. Set the recorder range on 1 and with the cooled syringe introduce a two-microliter sample into the chromatograph (The sample size must be chosen so that all peaks stay on scale). Keep all peaks on scale and run for twenty minutes to record any higher boiling impurities. The chromatogram consists of a series of peaks, following air and carbon dioxide.

4.4.1.5 Calculation. The percent of bromochlorodifluoromethane (BrClF_2C) shall be calculated as follows:

$$\% \text{BrClF}_2\text{C} = \frac{A(\text{BrClF}_2\text{C})}{A_s} \times 100$$

Where:

- a. $A(\text{BrClF}_2\text{C})$ = Area (in sq. cm) of bromochlorodifluoromethane peak multiplied by recorder range setting.
- b. A_s = sum of the relative peak areas

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4.4.2 Acid Halides. A large sample shall be evaporated in the presence of a small amount of crushed ice - distilled water slurry. The solution is titrated for acid halides with standardized sodium hydroxide.

4.4.2.1 Reagents.

a. Sodium hydroxide, 0.01N solution: Dissolve 0.40 grams of carbonate free sodium hydroxide in recently boiled distilled water in a 1000-ml volumetric flask. Cool and dilute to the mark. This solution shall be standardized against reagent grade potassium acid phthalate.

b. Methyl red indicator, 0.1% solution.

4.4.2.2 Procedure. Place 10-ml of a crushed ice-distilled water slurry in a 250-ml glass stoppered Erlenmeyer flask and add 50 grams of bromochlorodifluoromethane to the slurry. Place the stopper in the flask loosely and swirl the flask gently from time to time until the ice is completely melted. Add one drop of methyl red indicator and if a reddish color remains, titrate to a yellow endpoint with 0.01N sodium hydroxide solution.

CAUTION - Perform the above procedure in a hood.

4.4.2.3 Calculation. The ppm acid halides, as HBr, shall be calculated as follows:

$$\text{ppm Acid Halides (as HBr)} = \frac{\text{ml NaOH} \times N \text{ NaOH} \times 0.0809 \times 10^6}{\text{weight of sample (grams)}}$$

4.4.3 Free Halogen. The free halogen shall be treated with an excess of potassium iodide and the iodide liberated is titrated with sodium thiosulfate solution.

4.4.3.1 Reagents

a. Sodium thiosulfate, 0.01N solution: Prepare a 0.1N solution by dissolving 25 grams of sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$) and 0.5 gram of sodium carbonate in one liter of distilled water. Standardize against 0.1N potassium dichromate solution. From this 0.1N solution of sodium thiosulfate, prepare a 0.01N solution: Pipet a 10-ml aliquot of the standard 0.1N sodium thiosulfate solution into a 100-ml volumetric flask, fill to the mark with distilled water and mix. Prepare the 0.01N sodium thiosulfate solution fresh daily.

b. Sulfuric Acid, 1:4 solution in water.

c. Potassium iodide, 10 percent solution in water.

d. Starch indicator.

4.4.3.2 Procedure. Pour 100 ml of 10 percent potassium iodide solution into a 250-ml Erlenmeyer flask. Add 10-ml 1:4 sulfuric acid and 1-ml of starch solution. Bubble 100 grams of bromochlorodifluoromethane vapor through the potassium iodide solution. Titrate any liberated iodine with standard 0.01N sodium thiosulfate solution. Run a reagent blank along with the sample.

4.4.3.3 Calculation. The ppm of free halogen, as bromine, shall be calculated as follows:

$$\text{ppm Free Halogen (as Br)} = \frac{(A - B) \times N \text{ Na}_2\text{S}_2\text{O}_3 \times 0.0799 \times 10^6}{\text{weight of sample in grams}}$$

Where:

a. Ml $\text{Na}_2\text{S}_2\text{O}_3$ for sample

b. Ml $\text{Na}_2\text{S}_2\text{O}_3$ for blank

4.4.4 Non-Volatile Residue. A large volume of bromochlorodifluoromethane shall be evaporated and the residue weighed.

4.4.4.1 Procedure. Add 100-ml of bromochlorodifluoromethane to a weighed evaporating dish. Allow the sample to evaporate to dryness in a hood. After evaporation is complete, dry for 15 minutes in a drying oven at 105°C. Cool the evaporating dish in a desiccator and reweigh. The gain in weight (residue) is expressed in grams per 100-ml.

4.4.5 Suspended Matter and Sediment. Examine visually for any suspended matter or sediment.

4.4.6 Color. A sample of bromochlorodifluoromethane shall be compared with color standards (ASTM D1209 or D2108).

4.4.6.1 Reagents

a. Platinum - Cobalt Stock Solution. Dissolve 1.245 grams of potassium chloroplatinate (K_2PtCl_6) and 1.000 grams of cobaltous chloride ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$) in water. Add 100 ml of concentrated hydrochloric acid and dilute to 1 liter with water. This solution has a color of 500.

b. Platinum-Cobalt Standards. Prepare color standards having a color of 5, 10, 15 by diluting 1.0, 2.0, 3.0 ml of above solution in Nessler tubes to 100 ml with water.

4.4.6.2 Procedure. Pour the sample into a cold, clean, dry Nessler tube and compare with the standard by looking down through the Nessler tube against a white background. The number of the standard that most nearly matches the sample is reported as the color.

4.4.7 Boiling Point. The bromochlorodifluoromethane shall be allowed to boil, under reflux, until the temperature becomes constant. This constant temperature is the observed boiling point.

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4.4.7.1 Apparatus:

- a. Boiling Point Apparatus as shown in Figure I or Cottrell Molecular Weight Apparatus.
- b. Refrigerant system capable of cooling to a -25°C or lower.
- c. Barometer. An accurate barometer that can be read or converted to mm Hg.

4.4.7.2 Procedure. Cool the boiling point apparatus thoroughly by allowing the refrigerant to flow through the condenser for 15 to 20 minutes before addition of sample. Add bromochlorodifluoromethane through the top of the condenser, slowly, until the sample is just above the partition plate of the heating pot section. Replace the drying tube in the condenser opening and slowly increase the powerstat setting until a good reflux is obtained. The refluxing should continue until a constant temperature is reached. Note the barometric pressure and record the constant temperature to the nearest 0.1°C as the observed boiling point.

4.4.7.3 Calculation. The observed boiling point shall be corrected to the boiling point at 760 mm Hg as follows:

$$\text{B.P.}^{\circ}\text{C} = \text{Obs. B.P.}^{\circ}\text{C} + 0.00012 (760 - \text{Obs. Bar. Pressure})(273 + \text{Obs. B.P.}^{\circ}\text{C})$$

4.4.8 Moisture. Analysis shall be in accordance with Federal Test Method 791, Method 3253.

4.5 Rejection. Failure of any container to conform to the requirements of this specification shall be cause for rejection of the entire lot.

5. PREPARATION FOR DELIVERY

5.1 Packaging.

5.1.1 Levels A and C. Unless otherwise specified, bromochlorodifluoromethane shall be delivered in cylinders of 35- and 200-pound capacity conforming to Interstate Commerce Commission specification ICC 4B300 or ICC 4BA300, and in 2600-pound tanks conforming to Interstate Commerce Commission Specifications ICC 106A500 or ICC 110A500W. The contractor shall assure that containers are free from contamination and are suitable for shipment and storage.

5.2 Marking. In addition to any special marking required by the contract or order, containers shall be marked in accordance with Military Standard MIL-STD-129. Filled cylinders shall be color coded in accordance with MIL-STD-101.

6. NOTES

6.1 Intended Use. The bromochlorodifluoromethane covered by this specification is intended for use as a fire extinguishing agent.

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6.2 Ordering Data. Procurement documents should specify the following:

- a. Title, number and date of this specification.
- b. Quantity of bromochlorodifluoromethane desired.
- c. Type and size of containers required.

6.2.1 Bromochlorodifluoromethane shall be purchased by weight, the unit being an avoirdupois pound.

6.3 The percentage of bromochlorodifluoromethane may be determined as specified herein or by a method acceptable to the procuring agency.

6.4 The manufacturer of Chromosorb-R is Johns-Manville.

Custodian:
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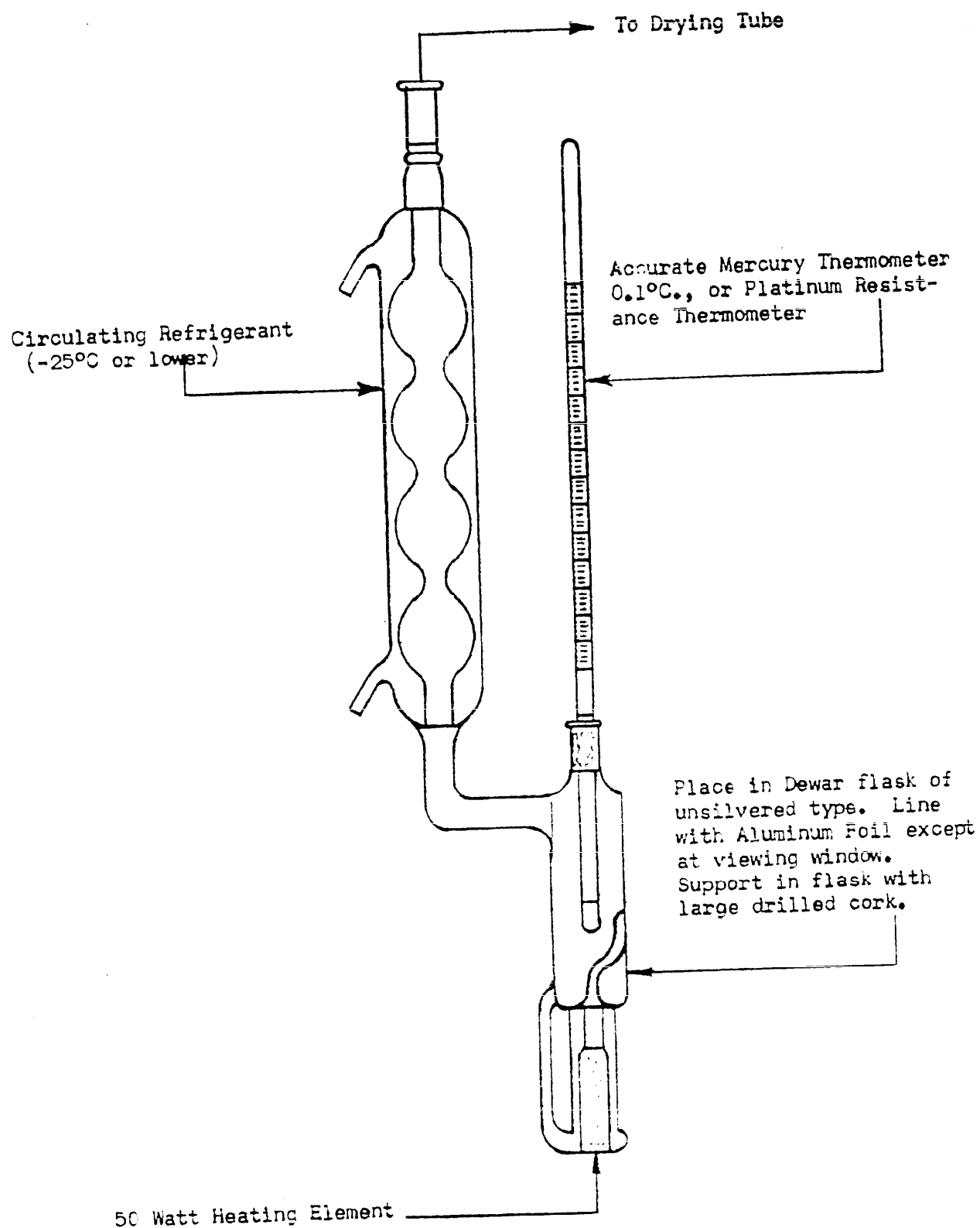


FIGURE I - BOILING POINT APPARATUS

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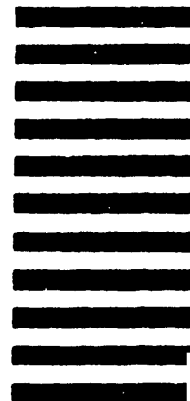
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c. Reason/Rationale for Recommendation:

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